Fast Screening of Pesticides and Endocrine Disrupters Using the Agilent 6890/5973N GC/MSD System, Part II

Application

Gas Chromatography
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Authors

C. Kai Meng
Agilent Technologies, Inc.
2850 Centerville Road
Wilmington, Delaware 19808-1610
USA

Michael J. Szelewski
Agilent Technologies, Inc.
2850 Centerville Road
Wilmington, DE 19808-1610
USA

Abstract

Agilent Technologies' new, fast GC/MSD method can significantly speed up the screening of pesticides. Agilent's GC Method Translation software (available free from the Agilent Technologies Web site, http://www.chem.agilent.com/cag/servsup/usersoft/main.html#mxlator) was used in developing the new method based on the standard 42-min method. A 15 m × 0.25 mm × 0.25 µm Agilent HP-5MS column was used to increase analysis speed up to fourfold. The time savings were implemented in increments (down to 10.5 minutes) to verify the predictability of scaling and the effect of scaling on the signal-to-noise ratio.

Key Words

RTL, pesticide, environmental, screening, fast GC, method translation, 5973, 6890, MTL

Introduction

Analysts want faster analyses to improve laboratory productivity. Often, when speeding up GC methods, an analyst will trade resolution for increased analysis speed. This loss of resolution can complicate peak identification, even with a mass selective detector (MSD).

Agilent Technologies has developed new techniques to solve the peak identification problem based on Agilent's retention time locking (RTL) and a new mass spectral library that contains the locked retention times and characteristic ions for 567 of the most common pesticides and endocrine disrupters of concern worldwide. A GC/MSD method was developed based on the standard 42-min method[1] to screen for all 567 of the most common analytes. A specific combination of column stationary phase, carrier gas flow rate, and oven temperature programming is required to lock all the compounds to an expected retention timetable[2]. Compound identification based only on spectral searching alone is difficult when analyzing extracts containing significant sample matrix content because of overlapping peaks and noisy baselines.

The new screening tool, integrated within Agilent's ChemStation for MSD, searches for all 567 compounds. It first checks and integrates four characteristic ions within the expected time window and then prints a report showing "hits" and "possible hits" (ratios of characteristic ions that do not match the expected values in the library within specified limits).

In Part I of the MSD fast screening application brief[3], a 10 m × 0.1 mm × 0.1 µm Agilent HP-5 column was used to increase analysis speed up to fourfold. In this application brief, a 15 m × 0.25 mm × 0.25 µm Agilent HP-5MS column was used. The faster methods were scaled exactly as predicted by using a combination of Agilent's method translation (MTL) and RTL software. Because scaling was exact, these faster methods can be used with precisely-scaled pesticide libraries, making the screening process even more powerful and adaptable to individual needs.

Experimental

The GC method translation software tool was used to find operating conditions for the faster methods. Figure 1 is a screen capture of MTL software data entry showing the original conditions and the new chromatographic conditions for a fourfold speed gain. The column flow rate, which is helpful to avoid exceeding MSD pumping capacity[4], also is found in the table. In this study, a turbo pump was used, which could handle the 3.8 mL/min carrier flow. The program also determined the required column head pressure and corresponding oven ramp. The Agilent 6890 GC fast oven option (220/240V in the U.S.) was required for the faster oven ramp used in this study.

General chromatographic conditions are listed in table 1. The standard used was a mixture of 26 pesticides at 10 ppm. A 15 m × 0.25 mm × 0.25 µm Agilent HP-5MS column (part number 19091S-431) was used. The head pressure determined by the method translation software (18 psi) was used as the starting point for retention time locking. The column head pressure required to lock retention times of the compounds to the library (the original retention time divided by 4) was determined using the automated RTL process integrated within the Agilent ChemStation for MSD.

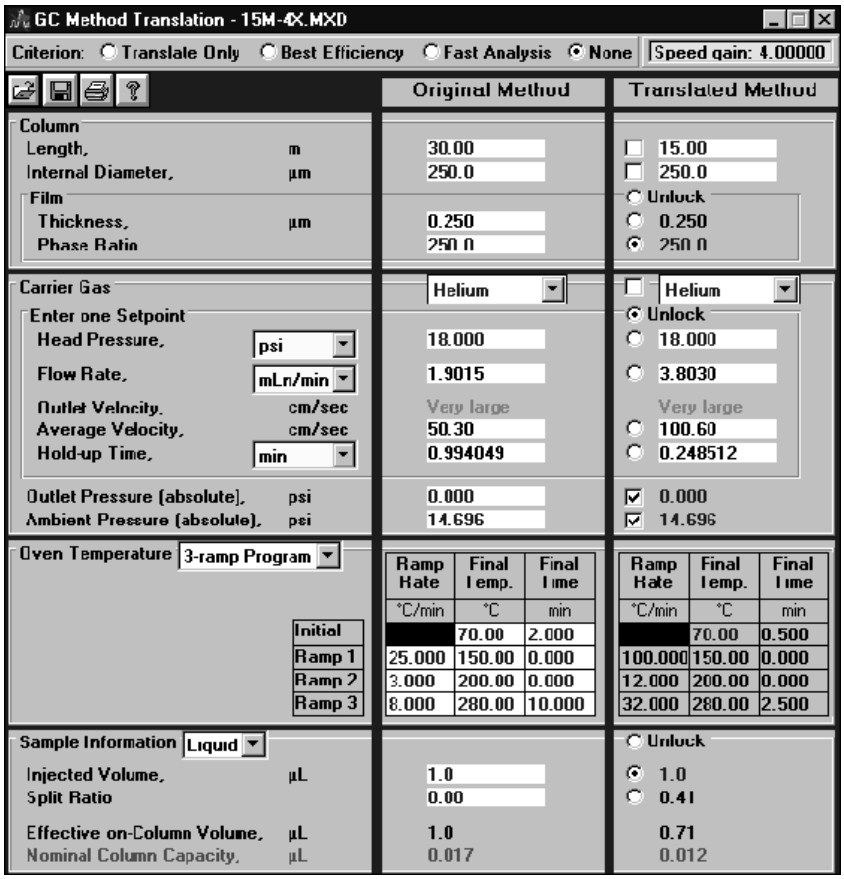

Figure 1. Screen capture showing the method translation (MTL) software data entry used in a 4X speed gain translation.

This process (first translate the method then lock the retention times) was repeated for the 2.5X time reductions.

Figure 2 shows the results of the shortened analysis times. The three chromatograms look extremely similar, except that the time axis is scaled proportionally. Because MTL followed by RTL scales methods very precisely, scaled screening libraries for corresponding time reductions can be obtained by dividing the retention times in the library by the speed gain (which does not have to be an integer). Using the same injection method (1-µL splitless), the peak heights of the faster runs were twice those from the original

Table 1 Chromatographic Conditions

Speed	**Onefold**	**Two and a half fold**	**Fourfold**
GC	110 V	220/240 V	
Column	30 m × 0.25 mm × 0.25 µm HP-5MS (P/N 19091S-433)	15 m × 0.25 mm × 0.25 µm HP-5MS (P/N 19091S-431)	
Injection mode	Splitless	Splitless	
Column head pressure	18.0 psi	5.74 psi	18.0 psi
Column flow (mL/min)	1.9	1.49	3.8
Inlet control mode	Constant pressure	Constant pressure	
Carrier gas	Helium	Helium	
Injector Temp.	250 °C	250 °C	
Oven Temp.	70 (2 min)	70 (0.8 min)	70 (0.5 min)
Ramp 1	25 °C/min	62.5	100
	150 (0 min)	150 (0 min)	150 (0 min)
Ramp 2	3 °C/min	7.5	12
	200 (0 min)	200 (0 min)	200 (0 min)
Ramp 3	8 °C/min	20	32
	280 (10 min)	280 (4 min)	280 (2.5 min)
Oven equilibration	2 min	2 min	
Injection volume	1 µL	1 µL	
Liner	5183-4647	5183-4647	

MS Conditions (Turbo pump)			
Solvent delay	3 min	1.44 min	0.9 min
Tune file	Atune.u	Atune.u	
Low mass	35 amu	35 amu	
High mass	500 amu	450 amu	
Threshold	150	250	
Sampling	2	2	1
Scans/sec	3.15	3.50	6.54
Quad Temp.	150 °C	150 °C	
Source Temp.	230 °C	230 °C	
Transfer line Temp.	280 °C	280 °C	
Acquisition mode	Scan (EI)	Scan (EI)	

analysis. A faster oven ramp and the shorter column made the peaks narrower and higher, so an improvement in the signal-to-noise ratio is realized with the faster methods.

Conclusion

The highly accurate and reproducible pressure and temperature control of the Agilent 6890 GC allows precise scaling of the standard 42-min GC/MSD pesticide method. Run time was shortened to 10.5 minutes using a fast oven ramp rate and a 15-meter, 250-micron column. The combination of MTL and RTL facilitated scaling and yielded exact scaling. RTL libraries can be scaled accurately to correspond to the faster analyses.

References

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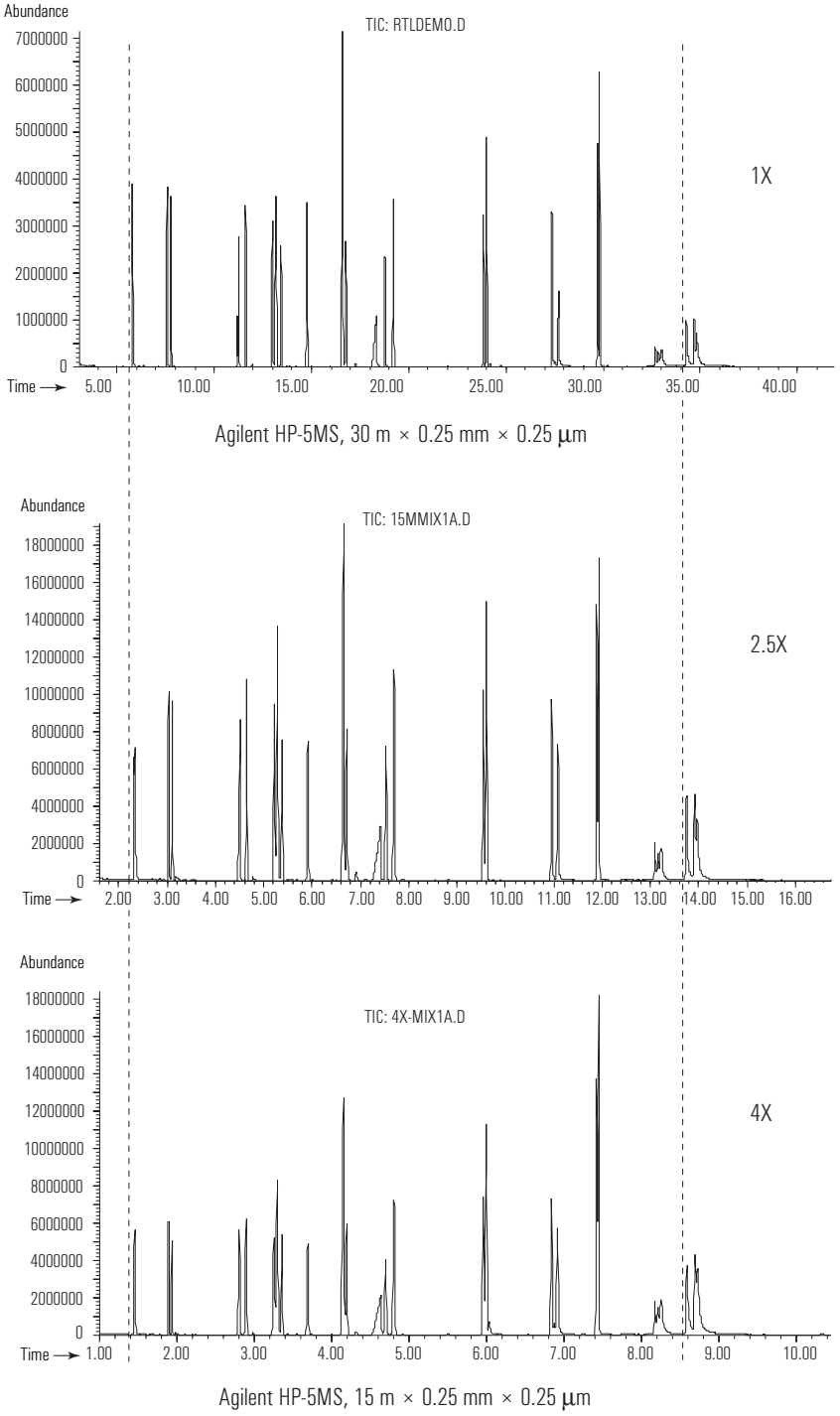

Figure 2. The TICs of the 2.5X and 4X speedups. The standard analysis (1X) was 42 minutes long.

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